International Journal of Agricultural
Science and Research (IJASR)
ISSN(P): 2250-0057; ISSN(E): 2321-0087
Vol. 9, Issue 3, Jun 2019, 323-332

EXTRACTION AND CHARACTERIZATION OF NANOFIBRILLATED CELLULOSE FROM BANANA PSEUDOSTEM BY STEAM EXPLOSION

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ABSTRACT

Primary goal of the current study is to extract the nanocellulose or nano fibrillated cellulose(NFC) from banana variety "poovan" pseudo-stems via steam explosion method. Steam explosion combined with bleaching, alkali and acid treatments of the banana fibres was resulted to be effective separation of fibrils from raw banana fibre bundles. The resultant product was well defibrillated cellulose with nano range. The use of novel bio-based materials having improved mechanical strength and toughness along the low weight percentage, biodegradable nature, and renewability. Morphological, Functional, Chemical and Spectroscopic properties of the NFC samples were analyzed and results were stated that the separation of nanofibrilis occurs after completion acid hydrolysis and mechanical stirring. The extracted nano fibrils from banana pseudostem possess improved properties for various advanced nanotechnological applications.

KEYWORDS: Nano Fibrillated Cellulose (NFC), Steam Explosion & Acid Hydrolysis

Received: Apr 27, 2019; **Accepted:** May 17, 2019; **Published:** Jun 01, 2019; **Paper Id.:** IJASRJUN201945

Original Article

1. INTRODUCTION

Cellulose is defined as a complex carbohydrate with a chemical formula of $(C_6H_{10}O_5)_n$ is made up of glucose sub units, and is important in the manufacturing of numerous product(Chirayil, Mathew, & Thomas, 2014). Cellulose is abundant and widely distributed natural polymers (Klemm et al., 2011). It is estimated that more than 1 ×1011 tonnes of banana being produced each year in our planet. One of the important recent developments in the area of bio-based materials is the production of nano cellulose (Kim, Stannett, & Gilbert, 1976). Because of increasing environmental safety, interest in the use of agricultural wastes, and cellulose based novel bio materials have gained greater potential in the past few decades(Gañán, Zuluaga, Restrepo, Labidi, & Mondragon, 2008).

Nano cellulose is defined as the nano structured cellulose. It may be either cellulose nanofibers (CNF), also called micro fibrillated cellulose (MFC) or nano fibrillated cellulose (NFC) based on size of the fibre. NFC exhibits greater mechanical strength, stiffness and is relatively abundant, biodegradable(Siró & Plackett, 2010). NFC have a larger surface area and improved mechanical properties (Bhatnagar & Sain, 2005), and it can be investigated for use in nanocomposites, coatings and films for packaging.

Agricultural waste is also an abundant resource of cellulose. (Lee, Hamid, & Zain, 2014). Exploitation of the agricultural waste will be significantly beneficial to the environment and bring additional profits to farmers (Chen, Wu, Huang, Huang, & Ai, 2014). Banana is important fruit crop cultivated in tropical parts of the world. The major component of the banana plant cell wall is made up of cellulose. Nendran peduncle fiber (60.27%) has highest cellulose content followed by pseudostem fibre of the Nendran (59.23%). Basarai, Lokhandi, Robusta, Shreemanti and Grand Naine are some of the available types of banana plant in India (Mukhopadhyay, Fangueiro, Arpac, & Şentürk, 2008). Cellulose obtain from such banana plants can be used for production of micro cellulose, as well as nano.

Steam explosion operates with high temperature and pressure, leads to cracking down the cell wall components of plants. It can then improves the degradation of hemicelluloses to glucose or xylose (Mosier et al., 2005). Steam explosion is found to be an effective method for the complete separation of nano fibrils from banana pseudostem(Cherian et al., 2008). High pressure steaming followed by rapid depressurization is called as steam explosion. (Cherian et al., 2010).

The study describes the efficient extraction of nano fibrillated cellulose (NFC)from banana pseudostem using steam explosion, bleaching, alkali and acid treatments followed by mechanical stirring. The Morphological, physico-chemical and functional properties of the synthesized NFC were studied using FT-IR, Scanning electron microscope (SEM), Transmission electron microscope (TEM).

2. MATERIALS AND METHODS

2.1. Materials

Fresh banana pseudostem variety "poovan" was collected from the orchard of Tamil Nadu Agricultural University (TNAU), Coimbatore. List of Chemicals used for NFC extraction was Sodium hydroxide(NaOH), Acetic acid, Sodium hypochlorite and Oxalic acid.

2.2. Isolation of Nanofibrillated Cellulose (NFC)

The inner most portion of the banana pseudostem was chopped and used for effective fibre extraction. Prior to fibre extraction, the Initial weight of the pseudostem was recorded. The fibre was extracted using a **Banana fibre extractor** and the extracted fibre were allowed to dry under sunlight for about 24 hour. Then the fibre were chopped into small pieces in the range 5-10cm. Chopped fibres were treated with 2% NaOH with a ratio of 1:10 and kept in an autoclave 120ºC, 20lb pressure for about 1hour**(Alkali treatment**). The fibres were washed with distilled water for the complete removal of alkali from the fibre. After steam explosion, bleaching was done with the mixture of NaOH and CH_3COOH (2.7 and 7.5g) and Naoclin the ratio of 1:3.Repeated the bleaching stem for better removal of yellow layer from banana fibre. Bleached fibres were treated with 10% oxalic acid in autoclave at a pressure of 20lb. The pressure was released immediately. The **Acid hydrolysis** step was repeated eight times for better digestion and defibrillation. The fibres were taken out from autoclave and washed with distilled water to make them free from acids(Abraham et al., 2011). The processed nano fibrils were suspended in water and kept stirring with a **mechanical stirrer** at 1500 for about 16hrs.

2.3. Characterization

2.3.1. Fibre Yield & Recovery and Chemical Analysis

Fibre recovery percentage is defined the amount of fibre that can be extracted from the fresh banana pseudostem. The fibre recovery percentage and fibre yield of the banana pseudostem were estimated by using this formula (Gopinathan, Subramanian, Paliyath, & Subramanian, 2017).

$$\text{FibreRecovery (\%)} = \frac{\text{Initial Weight of the extracted fibre (g)}}{\text{Initial weight of pseudostem (fibre extractable)}} \times 100$$

$$\text{Fibre yield (\%)} = \frac{\text{Weight of the fibre after drying (g)}}{\text{Initial Weight of the extracted fibre (g)}} \times 100$$

According to ASTM standards, the Chemical constituent of raw, steam exploded and bleached fibres were determined. The percentage cellulose, hemicellulose, lignin and moisture content of the fibres were calculated.

2.3.2. Fourier Transfer-Infrared (FT-IR) Spectroscopy

FT-IR analysis of the Nano fibrillated cellulose was performed on Jasco FT-IR 6800 spectrometer. About 1.0 mg of fibre was used for this analysis. The chemical composition was examined using FT-IR on different stages of fibre extraction.

2.3.3. Scanning Electron Microscopy (SEM)

Morphological analysis of Nano fibrillated cellulose was done by scanning electron microscopy. SEM images of NFC surface were taken using scanning electron microscope in the Department of Nano Science and Technology, TNAU, Coimbatore. Prior to SEM analysis, the fibre samples were coated with gold by using a plasma sputtering apparatus.

2.3.4. Transmission Electron Microscopy (TEM)

TEM images of nanofibrillated cellulose were taken in a transmission electron microscope with an accelerating voltage of 120 kV. A diluted suspension of nano fibrillated cellulose was coated on the surface of carbon-coated grid for imaging in TEM.

3. RESULTS AND DISCUSSIONS

Steam explosion is one of the effective methods in NFC extraction. In this method alkali treatment, bleaching, acid hydrolysis and mechanical stirring were carried out to produce a nano fibrils from raw fibre of the banana pseudostem. Alkali treatment with NaoH effectively remove the lignin, wax, hemicellulose and oils from outer layer of the fibre cell wall and defibrillated the cellulose into short length cellulose crystals. Then the fibres were subjected to steam explosion with high pressure and rapid depressurization, causing the fibres to get disintegrated. The structure of the fibres were get softened after this process. Bleaching is the important step to completely eliminate the remaining cemented/binded materials from the steam exploded fibre. Lignin and hemicellulose percentage was gradually decreased from raw fibre to bleached fibre. The samples were further down sized using high energy mechanical milling/stirring. Mechanical stirring process mainly used for effective size reduction and better dispersion of nano cellulose.

3.1. Fibre Yield & Recovery and Chemical Analysis

The fibre yield and recovery percentage of banana pesudostem were recorded using the formula mentioned in 2.3.1.

Table 1: Fibre Yield and Recovery Percentage of Poovan Banana Fibre

No.	Wt. of Banana Pesudostem (kg)	Recovery (g)	Recovery (%)	Fibre Yield (g)	Fibre Yield (%)
1.	15	432	2.88	18	4.1
2.	13	367	2.82	12	3.2
3.	10	290	2.90	9	3.1

Table 2: Components of Fibre in Different Processing Stages

No.	Processing Stage	Cellulose %	Hemicellulose %	Lignin %
1.	raw banana fibre	63.5	18.7	5.2
2.	steam exploded fibre	85.1	5.2	2.4
3.	bleached & acid treated fibre	92.0	1.01	0.3

Table 2 describes the various chemicals in the banana fibre at different levels of processing. Crystallinity percentage of cellulose get increased with the treatment of NaOH, steam explosion, bleaching and acid hydrolysis. (Klemm, Philpp, Heinze, Heinze, & Wagenknecht, 1998). Initially the hemicellulose and lignin percentage of raw banana fibre was relatively high. After different processing stages, hemicelluloses and lignin percentage was gradually decreased. It shows that, the percentage of pure cellulose content was increased due to steam explosion, acid and alkali treatments

3.2. Fourier Transfer Infrared Spectroscopy (FT-IR)

(Erdtman, 1972) reported that, the banana fibres are mainly composed of aromatics, ketones, esters, alkanes and alcohols, with different oxygen-containing functional groups. Infrared transmittance spectra with main observed peaks of the fibre in different stages are shown in **Table 3**.

Table 3: FT-IR Analysis of the Studied Banana Fibers in Different Processing Stages

FT-IR(cm^{-1}) Spectra (%T)								
No	Samples	– OH Stretching	C–H Vibration	C O Stretching	Absorbed Water	C–H Stretching	Aromatic Ring Vibration of Lignin	C–C Stretching
1	Raw Banana fibre	3600	2361	1968	1640	1320	1240	1031
2	Bleached and acid treated	3594	2361	-	1642	1368	-	1031
3	Nano fibrillated cellulose	3599	2369	-	1648	-	-	1021

Lignin have significant peaks in the region of 1200–1300 cm^{-1},aromatic C C, exhibits the characteristics peaks in the region of 1830 cm^{-1} and 1730 cm^{-1}(Reddy & Yang, 2005). The –OH bending of adsorbed water at 1640 cm^{-1} was shown in **Table 3**. Peak in the region of 1968 cm^{-1} corresponding to the raw banana fibres due to the presence of C O linkage, which is a significant peak of lignin and hemicellulose, and its completely absent in bleached fibre and nanofibrillated cellulose. It shows that the minimum or no hemicellulose and ligin content in final NFC. From the functional analysis of FT-IR showed, that the percentage of cementing materials get reduced with the process of steam explosion and chemical treatment.

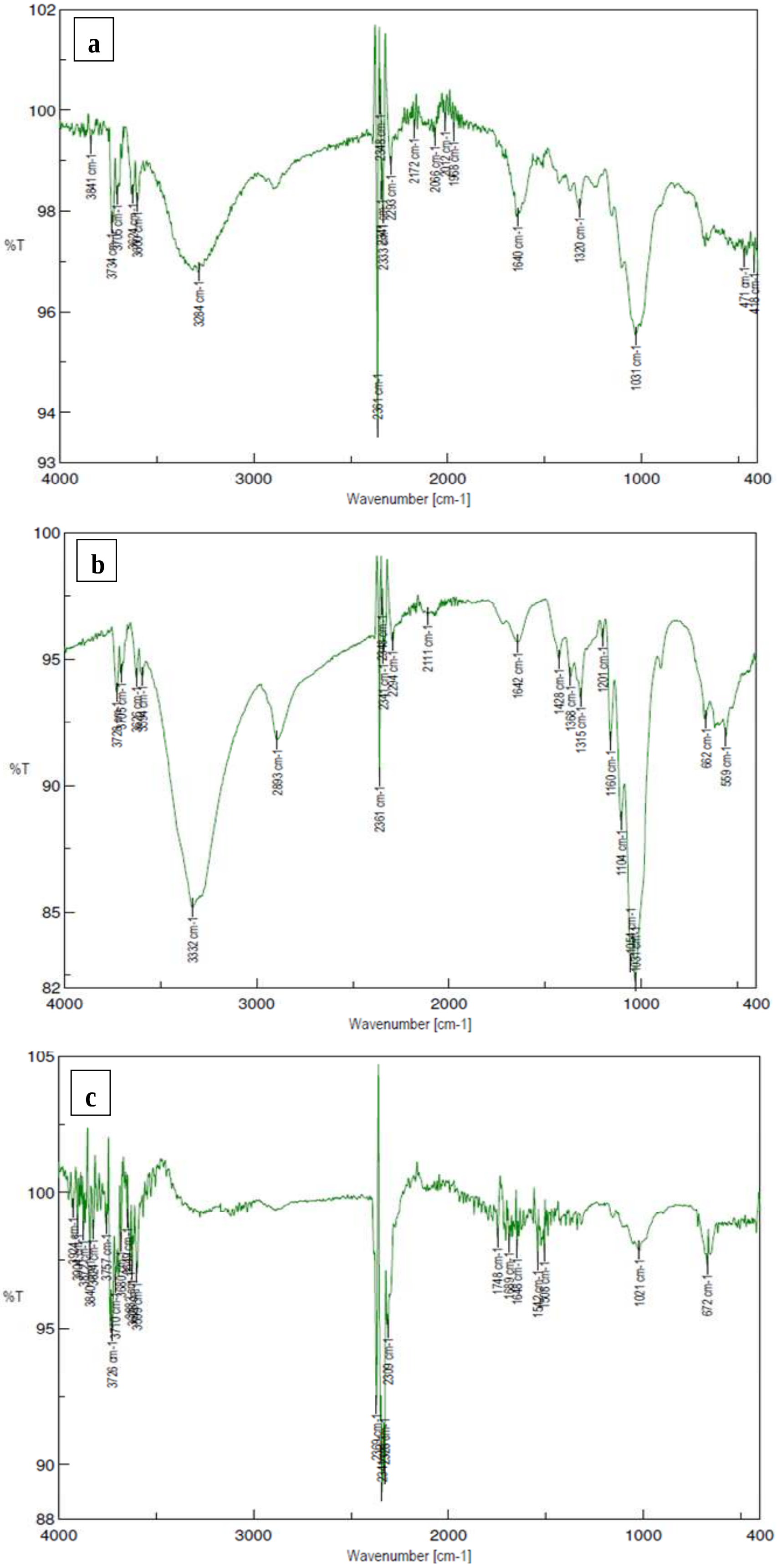
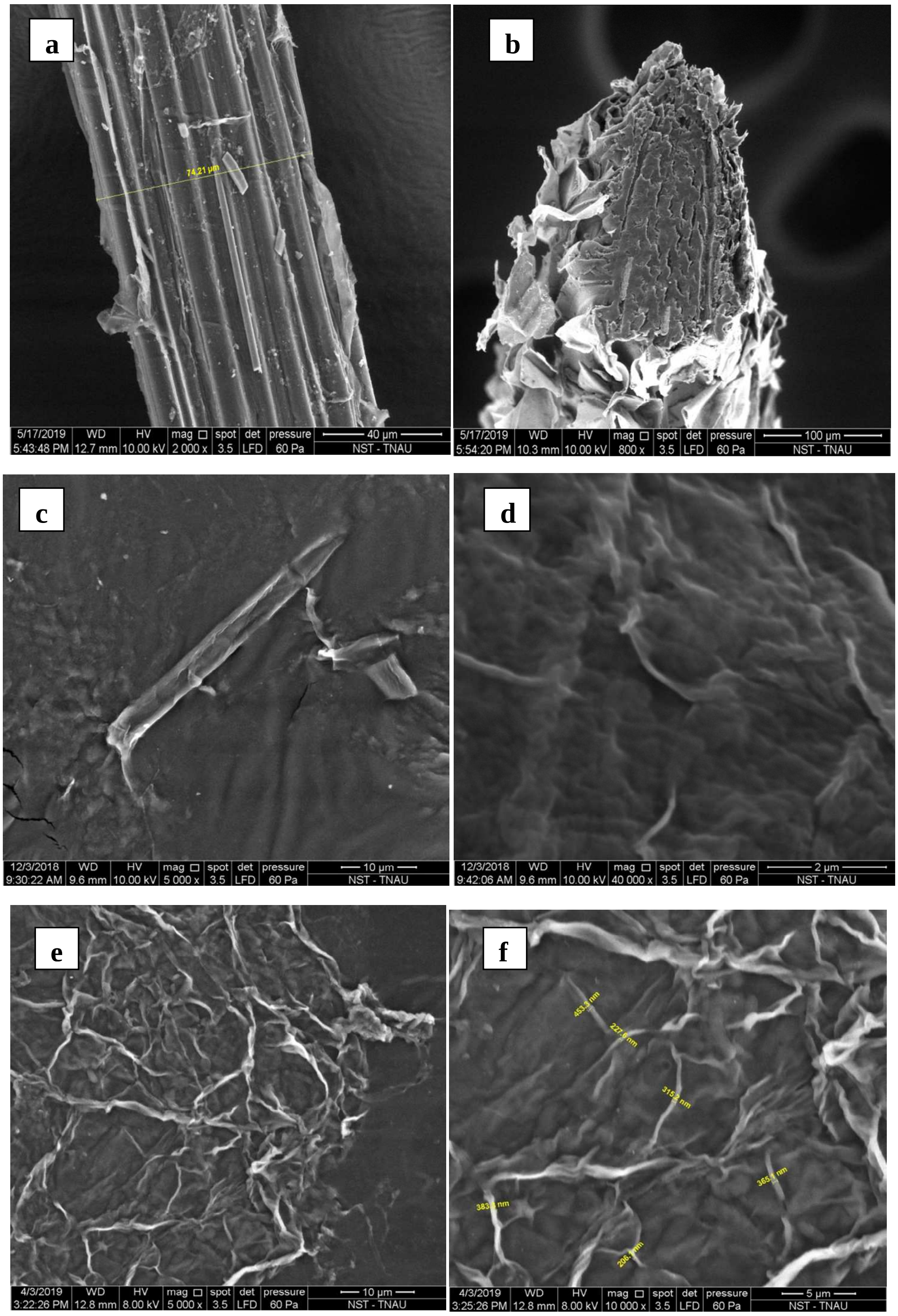


Figure 1: FTIR Images of a. Raw Banana Fibre, b. Bleached Fibre, c. Nanofibrillated Cellulose

3.3. Scanning Electron Microscopy (SEM)

The surface morphology of the banana fibers was imaged under SEM **(Figure 2)**. As shown in **(Figure a & b**) raw banana fibre shows more cementing and concreting material on the cell wall and it exhibits rough surface due to the presence of lignin, hemicellulose and wax. **(Figure c & d)**, Micro fibrils were partially separated from the fibre bundles and defibrillation started at this stage. It concluded that the non cellulosic compounds were completely removed from the Fibre **(Figure e & f)**. Complete defibrillation occurred in the final stage of processing. Long and rough fibre bundles were disintegrated and it breakdown into small fragments of fibres with nano scale called Nano fibrillated cellulose.



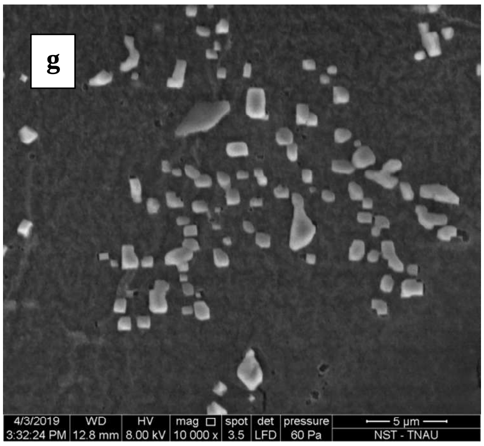
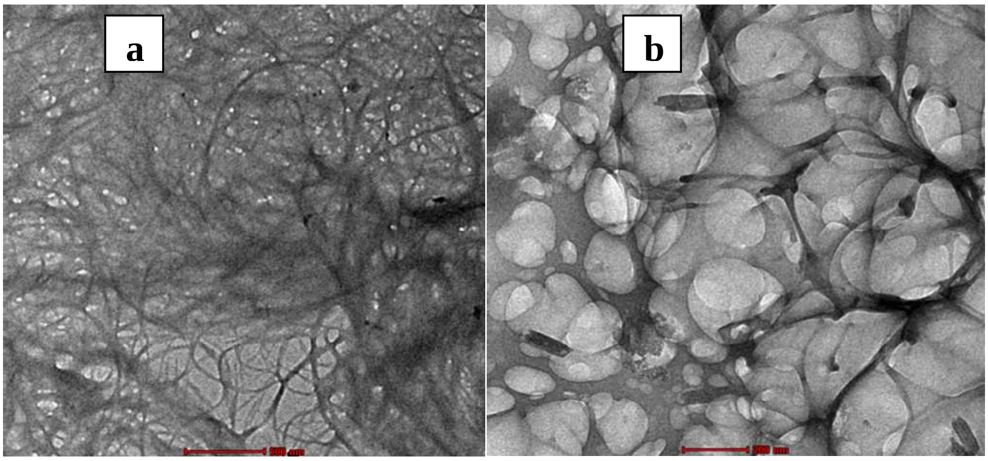


Figure 2: SEM Images of Banana Fibre at Different Stage g. Figure a& b. Raw Banana Fibre, Figure c & d. Bleached Banana Fibre, Figure e & f. Nano Fibrillated Cellulose, Figure g. Nano Crystals

3.4. Transmission Electron Microscopy (TEM)

The internal portion of the banana fibres was imaged by TEM **(Figure 3) (Figure a & b**) The TEM analysis of bleached banana fibre revealed that the bleached fibre having more agglomeration and incomplete defibrillation. In this stage, only the cementing non cellulosic impurities were removed. It shows that the minimum or no fibre bundles are present at this stage. The individual fibres were partially separated and it exhibits long thread like appearance (**Figure c, d & f**). The fibres were completely depolymerized and no agglomeration occurred. Individual fibres are clearly visible at this stage. Micro fibrils were cracked into nanocellulosic fibrils through mechanical stirring process. Individual fibres were measured and it could be in nano range. TEM analysis revealed that, the steam coupled chemical treatment was enhanced the extraction of pure crystalline nano fibrils from banana fibre. These NFC can exhibit better mechanical, thermal properties. So it can be used as a filler materials for many nano composites.



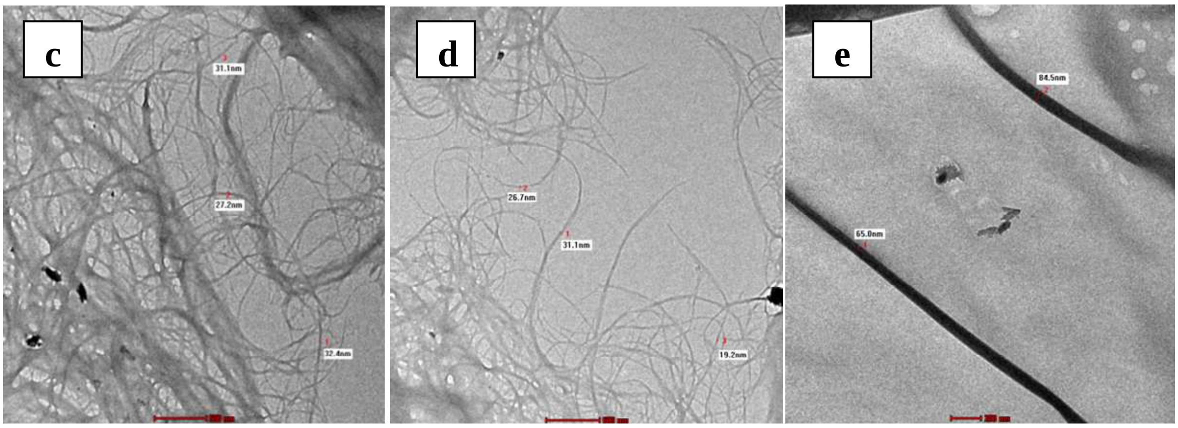


Figure 3: TEM Images of Banana Fibre at Different Processing Steps. Figure a & b. Bleached Fibre, Figure c & d. Nano Fibrillated Cellulose, Figure e. Individual Fibre (Defibrillation)

4. CONCLUSIONS

Cellulose nanofibrils from the banana pseudostem fibers exhibits improved morphological, chemical and crystalline properties. These nano fibrils can be used as a potential reinforcing component in the production of polymer nano composites. Steam coupled with alkali and acid treatment is the low cost and effective method in nano fibrils extraction. The percentage of pure cellulose components was increased with various processing stages. The structural, morphological and chemical properties of nano fibrils is more improved than the raw banana fibre. Studies concluded that the nano fibrils from banana pseudostem possess desirable properties that can be used for producing bio degradable nano packaging films. These results embarks a new journey to find a better packaging materials to reduces the post-harvest losses of fruits and vegetables. Eco friendly nano films from banana cellulose may be a better alternative for plastics to ensuring the environmental safety

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